ADVANCED MATERIALS AND CHEMICALS

Nanophase Technologies Corporation

Processing Nanoscale Materials for Large-Scale Production

The scientific community is in strong agreement that nanoscale materials, which are less than 100-billionths of a meter in size, exhibit superior chemical, mechanical, electronic, magnetic, and optical properties. When this project was funded in 1992, it was believed that this technology could increase U.S. market share in the diesel engine and ball-bearing industries. At that time, however, these nanocrystalline materials could only be produced in small quantities. Nanophase Technologies Corporation (NTC), with funding from the Advanced Technology Program (ATP), increased its capability to produce nanoscale materials by 25,000-fold and reduced its costs by 20,000-fold. These materials are now used in products as diverse as cosmetics, catalysts, and ceramics.

COMPOSITE PERFORMANCE SCORE

(based on a four star rating)

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Research and data for Status Report 91-01-0041 were collected during October-December 2001.

Commercial-Scale Production Requires Improved Processes

In 1992, nanocrystalline materials could only be produced in small quantities. To solve this problem, Nanophase Technologies Corporation (NTC) submitted a proposal to ATP and received funding for a three-year project. NTC sought to perform the research and development (R&D) necessary to enable the synthesis and processing of nanocrystalline ceramics on a commercial scale. The company's underlying strategy was to develop a multiuse technology that would provide a basis for future scale-ups of other nanoscale materials. NTC used its proprietary gas-phase-condensation (GPC) process as a foundation for the new technology. A unique feature of the GPC process is its ability to form stable and unique material structures.

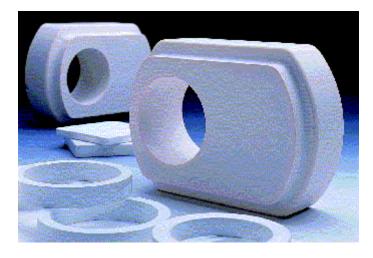
Manufacturing Techniques Limit Ceramics' Commercial Potential

Even though traditional, industrial-grade ceramics are suitable for general purposes, their use is limited by their low stress tolerances. The molecular breakdown of ceramics limits their marketability, because of either physical- or temperature-limiting characteristics.

Moreover, the machining costs of ceramic composites represent up to 95 percent of total production costs, thereby decreasing company profits. When this ATP project began, the chemical and physical properties of most materials used in industrial products did not yield the desired dimensional or stress tolerances. This shortfall resulted in weak components, including engine parts, bearing systems, electronics packaging, precision electrical and optical-fiber connectors, cutting tools, and sealing systems.

Previous methods of manufacturing nanocrystalline ceramics were unsuitable for commercial applications.

The manufacturing and industrial communities recognized that the benefits of using nanocrystalline ceramics could be significant. Previous methods of manufacturing nanocrystalline ceramics, however, were unsuitable for commercial applications and produced only 10 grams of material per day at a cost of \$1,000 per gram. NTC realized that the commercial potential of these materials would only be realized if they were synthesized in larger quantities (kilograms and upwards).



Commercialization of nanocrystalline ceramics has led to more resilient and cost-effective products.

NTC Advances Its Gas-Phase-Condensation Technique

Techniques to synthesize particles with dimensions of a few nanometers have existed since the 1980s. NTC proposed to use its proprietary GPC technique to produce particles of nanometer dimensions, often referred to as ultra-fine powder (UFP). This technique involves vaporizing a precursor (usually a metal), then rapidly cooling the vapor to condense out nanocrystals. A subsequent reaction (e.g., oxidation) is then possible. Traditionally, GPC has been performed using systems based on natural convection. These systems are difficult to scale-up, however, so they were only used in the laboratory. The NTC's proprietary GPC process is onetenth the size and cost of natural convection systems, but can produce materials at rates 100 times faster. NTC believed further development of these systems would establish a viable technique for generating gas-phasecondensed UFP on a commercial scale. NTC would lay the foundation for commercial-scale production of nanocrystalline materials by accomplishing the following goals:

- Scale up production of nanocrystalline ceramic powders already produced in laboratories, from gram quantities to kilogram quantities
- Extend the techniques to other industrially important ceramics
- Develop processing techniques to consolidate the powders into parts

- Comprehensively characterize the properties of the consolidated materials
- Develop a low-temperature, net-shape-forming technique

NTC Creates High-Volume and Low-Cost Ceramic Materials

NTC's successful ATP-funded R&D led to scaled-up production (100 tons per year) and reduced costs. In addition to ceramics, this new process also can produce nanoscale metal oxides, such as titania, that are nearly spherical, close to uniform in size, free of chemical residues, and loosely clustered. This enables the engineering of materials with specific attributes such as high strength or a particular color.

The successful scale-up and integration of these nanocrystalline ceramics on a commercial scale has led to more resilient and cost-effective products. Benefits of the new products include the following:

- Lighter weight
- Longer life span/service life
- Increased resistance to corrosion
- Reduced need for lubricants
- Increased temperature resistance

Although the steel and composite parts now used in end-use products are cheap and relatively effective, replacing them with ceramics will provide significant benefits. Not only will the benefits be realized at a national level, they also will extend to export markets, thereby positively affecting the U.S. trade balance.

ATP's Contribution Is Significant

"Although my company is still very small in revenue size," Robert Cross, the president and chief executive officer of NTC, stated after completion of the ATP project, "we are now the world's leader in the production and marketing of nanocrystalline materials for a wide range of important applications. It was not always that way, and it would not have been possible were it not for the support of the ATP."

In the early 1990s, before the ATP project, NTC had only two employees, limited funds, and no customer base. Buoyed by co-funding from ATP to explore this new process, as of 2001, NTC had 61 employees, a proven technology, and a 24-hour-a-day factory, as well as relationships with major corporations that are testing and selling its products. The three-year ATP project, which ended in 1995, led to the following measurable benefits:

- NTC achieved a 25,000-fold increase in its capability to produce nanoscale materials and a 20,000-fold reduction in costs.
- NTC nanocrystalline titania is now being used in cosmetic sunscreens and is more effective per unit weight compared with the titania in competing products. The demand for this product is expected to increase exponentially. The use of NTC nanocrystalline titania sunscreens was not anticipated at the onset of this project.
- NTC's tests of prototype mechanical seals made with these nanoscale materials demonstrate up to a 10-fold increase in service life, while prototype industrial catalysts are as much as four times more active.
- NTC nanocrystalline titanian coating protects automotive and carpet fibers from ultravioletinduced damage.
- NTC developed processes to fabricate near-netshaped ceramics and electronic substrates using alumina nanocrystalline materials.
- NTC developed printing inks for high-opacity jettable inks used in industrial markets.
- NTC-produced iron oxides are incorporated into cosmetic pigments.
- NTC produces nanocrystalline zirconia, ceria, and yttria for use in the manufacture of catalysts and near-net-shaped ceramics.

NTC also was able to secure private venture capital funds from multiple sources and received three patents from the U.S. Patent and Trademark Office.

NTC Has Sustained Its Accomplishments

To date, NTC has achieved the following competitive advantages:

- Received 3 patents directly resulting from the ATP project, with an additional 28 patents licensed or pending in the United States, Europe, and Japan.
- Improved its time-to-market for new products (i.e., a reduction from 18 to 24 months to 12 to 18 months).
- Increased its capital funding to sustain planned development activities and expand its manufacturing capabilities.
- Secured financing through an initial public offering (IPO) in November 1997.
- Maintained \$18.6 million in capital with no longterm debt as of September 30, 2000.

NTC also has identified and continues to target four markets that offer promising application for its products: healthcare, advanced ceramics, environmental catalysts, and transparent functional coatings.

Conclusion

As a result of the ATP project, NTC has succeeded in synthesizing and processing nanocrystalline ceramics for commercialization. For example, the development of a production reactor continues to benefit product lines and serves as the test bed for powder synthesis. NTC has solid financial backing from numerous sources and continues to increase its client base, which currently consists of more than 20 companies worldwide.

PROJECT HIGHLIGHTS Nanophase Technologies Corporation

Project Title: Processing Nanoscale Materials for Large-Scale Production (Synthesis and Processing of Nanocrystalline Ceramics on a Commercial Scale)

Project: To research and develop methods of processing nanoscale materials for commercial-scale production, with an emphasis on nanocrystalline ceramics. The successful scale-up and integration of these nanocrystalline materials on a commercial scale will provide more resilient and cost-effective products. Benefits of this achievement will affect products, such as steel ball bearings, mechanical seals, and the diesel engine, and industries, such as aerospace and fiber optics.

Duration: 7/1/1992-6/30/1995 **ATP Number:** 91-01-0041

Funding (in thousands):

Total \$3,953

Accomplishments: NTC successfully

demonstrated the applicability of nanoscale materials and developed methods to produce these materials on a commercial scale. The company accomplished the following:

- Achieved a 25,000-fold increase in its capability to produce nanoscale materials and a 20,000-fold reduction in costs.
- Incorporated NTC titania successfully into cosmetic sunscreens, offering increased protection levels and competitive costs at a more effective per-unit weight than competing products
- Signed an agreement to distribute NTC materials to more than 300 companies
- Completed an IPO in November 1997

NTC received the following patents for technologies related to the ATP project:

 "Method of making nanostructured materials" (No. 5,460,701: filed July 27, 1993, granted October 24, 1995)

- "A system for making nonstructured materials"

 (No. 5,514,349: filed August 4, 1994, granted May 7, 1996
- "Nanocrystalline materials"
 (No. 5,874,684: filed May 3, 1996, granted February 23, 1999)

Commercialization Status: NTC currently has a worldwide customer base in excess of 20 companies. The company has accomplished this market expansion based on its increased manufacturing capabilities and the sixmonth reduction in its time-to-market for new products. In March 1997, NTC signed an exclusive five-year contract with an undisclosed customer to provide nanoscale materials. NTC is currently providing nanoscale materials for a wide variety of applications including cosmetics, sunscreens, printing inks, and catalysts. The company has achieved these advances as a result of ATP's support during its infancy.

Outlook: Expectations for this technology and for the company are strong. The technology has been incorporated into commercial products that are being used extensively across several industries.

Composite Performance Score: * * * *

Number of Employees: Two employees at project start, 61 upon completion of status report

Company:

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